PROJECT NUMBER: 1720
PROJECT TITLE: Analytical Microscopy
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I. STUDIES IN MICROSCOPY

A. Objective: Examine the ultrastructure of selected cigarette papers in support of the low sidestream project.

B. Results:

Plans were finalized to study the ultrastructural changes (1) and the macro and micro changes of the paper cross section (2) that occur in low sidestream (LSS) cigarette papers as a function of temperature (1). The first study is a collaborative effort between Ken Maloney and Karen Sanders. The second study will be completed by Vicki Baliga.

An experimental LSS paper from Kimberly Clark made with small particle size chalk and monoammonium phosphate was examined using STEM. It contained Ca particles of very small size. The particles did not encase the fibers (3).

A 100% flax paper with no additives was examined as a control using STEM. The separation of flax fibers into smaller, individual fibrils could be seen (4).

C. Plans: Seven LSS papers will be analyzed by microscopy after heating to 3 temperatures plus ambient. Microscopic examination will include fiber and crystallite structure (1, 2).

II. RESPONSE TO ANALYTICAL REQUESTS

A. Objective: Provide analytical support to R&D.

B. Results:

In support of project QUICK two carbon rods were analyzed by EDS. One was received from T. Sanders and was described as a 'handwarmer' carbon rod having a "silver" paint on its outer surface. The inner and outer surfaces were analyzed for heavy metals (5). The only elements found on the inner surface were Si, K, and Ca. The outer surface elements were Al, K, and Ca. The "silver" paint was aluminum based. The second carbon rod was described as an extruded carbon rod bound with polyvinyl alcohol and then heated to drive off the PVA. EDS analysis was performed on both the inner and outer surfaces of this sample (6). Outer surface elements found were Mn, Si, S, P, K, Ca, Mg, and Cl. Inner surface elements were Al, Si, S, K, Ca, Ti, Mn, Fe, and P. The sample exhibited a great deal of elemental variation within and between areas analyzed.

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A red pellet of unknown composition and structure, located in the center of a smoked cigarette filter, was submitted by B. Fenner for analysis. EDS was completed on the inner and outer surfaces of the pellet. No elements above Na were detected. Microscopic examine of the pellet showed that it contained cavities filled with red material throughout the polymer matrix. The pellet also had an additional layer coating the outer surface (7).

A commercial Smoke-Check CO monitor was submitted by Gen Baker for EDS and structural analysis. The 2 sealed standards strips contained Ti, Ca, Si, and some Al. The 0.1 standard also had a small amount of Fe. These materials consisted of a fine granular matrix. The CO monitor strip contained Al as the support material with S, Ca, Cl, and Pd. The Al support matrix consisted of coarse, much larger particles than the standards strips (8).

A metal coupon with black particles was submitted for analysis by Duane Watson. The coupon had been placed above the stems in the ART pressure vessel. The black particles were separated from the metal coupon by sonicating the coupon in methylene chloride. The particles were paramagnetic and the majority of particles contained Fe, Cr, and Ni and were flat plate-like structures. Some were round and contained only Fe (9).

A new system has been established for incoming sample requests to the Analytical Microscopy group (10). This system will provide microscopists with better information about samples to be analyzed. It also provides a means of establishing an in-house data base (central file) as well as a mechanism of incorporating appropriate samples into a directory of microscopy on tobacco and tobacco related materials.

C. **References**:

1. Baliga, V., "Proposal - Paper Degradation Studies as a Function of Heat: I. Ultrastructural Characterization," memo to Richard Cox, October 27, 1987.
2. Baliga, V, "Proposal - Paper Degradation Studies as a Function of Heat: II. Ultrastructure of Fiber Cross Section and Void Volume," memo to Richard Cox, October 27, 1987.
3. Sanders, K., P.M. Notebook #8374, p. 155.
4. Baliga, V., P.M. Notebook #8412, p. 102.
5. Sanders, K., P.M. Notebook #8374, p. 147.
6. Sanders, K., P.M. Notebook #8374, pp. 148-149.
7. Baliga, V., Sanders, K., "Analysis of Red Filter Pellet," memo to R. Cox, October 15, 1987.
8. Baliga, V., P.M. Notebook #8412, p. 98.
9. Baliga, V, "Debris From ART Pressure Vessels," memo to Duane Watson, October 23, 1987.
10. Sanders, K., "Requests for Analytical Microscopy," memo to Distribution, October 9, 1987.

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